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RESEARCH ON INHIBITED N_2O_4

Fourth Quarterly Report

H. E. Dubb
J. Fisher
J. Lecce

September 1967

Rockeetdyne
A Division of North American Rockwell Corporation
Canoga Park, California

TECHNICAL REPORT AFRPL-TR-67-248

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Air Force Rocket Propulsion Laboratory
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Edwards Air Force Base, California
Air Force Systems Command
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FOREWORD

The research reported herein was supported by the Air Force Rocket Laboratory, Research and Technology Division, Edwards Air Force Base, California, Air Force Systems Command, United States Air Force, under Contract F04611-67-C-0008, with Ralph Fargnoli, lst/Lt/USAF, RPCL, serving as Project Monitor.

The work described covers the period 1 June through 31 August 1967. The Responsible Scientist for this program is Dr. Hubert E. Dubb of the Physical and Engineering Chemistry organization, which is managed by Dr. K. H. Mueller. The work was conducted by members of Physical Chemistry, supervised by Dr. A. E. Axworthy; Engineering Chemistry, supervised by Dr. W. Unterberg; Analytical Chemistry, supervised by Dr. V. H. Dayan; and Metallurgical Analysis, supervised by Mr. E. F. Green. The principal contributors to the program were Dr. Dubb, Mr. J. Fisher, Mr. J. Lecce, and Mr. G. Brull.

This report has been assigned the Rocketdyne report No. R-6831-4.

W. H. EBELKE, Colonel, USAF
Chief, Propellant Division

ABSTRACT

This program is concerned with extending the engineering evaluation of the new storable liquid oxidizer INTO which is NTO inhibited with 1 to 3 weight percent FNO_2.

Storability tests are being conducted on INTO at ambient temperature and at 70 C in aluminum and iron tanks of 10- to 18-gallon volume. Two 24-gallon titanium 6Al-4V tanks which were to be tested during this program failed while fluorine was being bubbled into them to form FNO_2. This indicates that INTO is not storable in titanium 6Al-4V when formed in situ.

Titanium 6Al-4V specimens were uniaxially stressed to 90,000 psi and stored in INTO at ambient temperature and at 70 C for 43 days. Stress corrosion failure did not occur. This test has been discontinued because of the results with the 24-gallon titanium tanks and with the small titanium bombs.

Small-bomb storability tests (20 milliliters) in A286 steel and in 250 maraging steel at ambient temperature and at 70 C are in their eighth month. Chemical analysis for FNO_2 content continues to indicate that INTO is storable in A286 steel and that it is probably not storable in 250 maraging steel. Titanium 6Al-4V was similarly tested for 7 months. The data indicate that INTO is definitely not storable in this alloy under the test conditions.

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INTRODUCTION

The use of nitrogen tetroxide (NTO) has been continually hampered by corrosion problems. Dry NTO is not a highly corrosive liquid when in contact with most common metals of construction, but moist NTO is extremely corrosive because of the formation of nitric and nitrous acids by the reaction of NTO with water.

It has previously been demonstrated under Contract AF04(611)-10809 (Ref. 1) that the addition of a fluorine oxidizer to NTO leads to a marked reduction of the nitric and nitrous acid content of the propellant with the concurrent production of HF. It has also been demonstrated (Ref. 1) that if the fluorine oxidizer is FNO_2, the resulting oxidizer system is storable at 70 C in passivated aluminum, stainless-steel, and nickel containers.

The present program consists of an extended engineering evaluation of inhibited nitrogen tetroxide (INTO), which is NTO containing 1 to 3 weight percent FNO_2 (1.4 to 4.2 mole percent). INTO is being evaluated with respect to its storability for 12 months at ambient temperature and at 70 C in large tanks of 10- to 18-gallon capacity (Task I), and at ambient temperature, 70 C, and 130 C in small bombs of 20-milliliter capacity (Task III). It has been evaluated at ambient temperature and at 70 C with respect to its effect upon the stress corrosion problem encountered when NTO is stored under pressure in titanium 6Al-4V tanks (Task II).

TASK I: LARGE-TANK STORAGE

SUMMARY

The objectives of this task are to investigate the storability of inhibited NTO (INTO) in tanks of approximately 10-gallon capacity, and to investigate the in situ formation of FNO_2 (the inhibiting agent) in the tanks. The original plan was to store six tanks, each fabricated of a different alloy, for a period of 12 months at ambient temperature. In addition, six identical tanks were to be stored at 70 C for the same period. Of the 12 tanks, 10 have been loaded with NTO; INTO has been produced in situ by fluorine addition. These 10 tanks are being stored under the required test conditions. The remaining two tanks, both titanium, failed during the process of fluorine addition.

EXPERIMENTAL

Early during this report period fluorine was in the process of being added to two 24-gallon 6Al-4V titanium tanks and one 10-gallon 2219 aluminum tank. These were the last three tanks to be loaded during this program. The 2219 aluminum tank was one which had undergone fluorine addition earlier in the program (Ref. 2) but was found to contain less than the required amount of FNO_2. This tank was therefore placed back on the fluorine loading system. The addition of fluorine to the tanks was performed essentially as previously reported (Ref. 2). In general, the fluorine addition procedure is as follows:

1. During the day, the tank ullage pressures are increased approximately 70 psi by fluorine addition.
2. The tanks are allowed to stand overnight with an ensuing ullage pressure drop presumably due to FNO_2 dissolution.

3. In the morning, the tanks are vented until a constant vapor pressure is observed (no decrease in ullage pressure upon further venting). Fluorine is again added during the day.

After approximately 13 pounds of fluorine had been added to the three tanks, the two spherical titanium 6Al-4V tanks failed during the night of 27-28 June (Fig. 1). As a result of the tank failures, approximately 20 to 30 pounds of INTO slowly leaked onto the concrete floor of the test facility. Safety procedures previously set up for such a contingency resulted in immediate detection and subsequent correction with minimum damage to equipment.

The spherical titanium tanks were mounted on square aluminum frames and thus were supported at four points of contact. Each tank failure occurred at a contact point where the weight of the tank was concentrated. Close visual examination of the tanks revealed that severe corrosion had occurred over the interior surface. The corrosion was more severe where the metal had been in contact with liquid INTO. There was evidence that the corrosion occurred at an accelerated rate (stress accelerated corrosion) at all of the load bearing points. The photograph (Fig. 1) appears to reveal corrosion of the outer surface but the apparent corrosion was caused by corrosion products from the interior of the tank being washed onto the exterior surface and drying there. A water rinse completely removed the apparent corrosion. The 2219 aluminum tank was not harmed. As a result of the tank failures and of the small-bomb storability tests with titanium 6Al-4V (Task III), it was concluded that INTO is not storable in this alloy.

During the period 14 to 20 June, the tanks already in storage were analyzed for FNO_2 content; the results of the analyses are presented in Table 1. The 1018 carbon steel, 18 percent Ni (250 grade) maraging

Figure 1. Photograph of Titanium 6Al-4V Tank After Failure

TABLE 1

INTO ANALYSES (MAY AND JUNE 1967)

Tank Material	Test Condition	Analysis Date	Mole Percent FNO_2
347 Stainless Steel	Ambient	15 May	2.6 Removed from Storage
347 Stainless Steel	70 C	15 May	2.9
		19 June	0
1018 Carbon Steel	Ambient	15 May	6.8
		15 June	6.6
1018 Carbon Steel	70 C	15 May	4.2
		15 June	0
18 Percent Ni Maraging Steel	Ambient	15 May	5.7
		14 June	6.0
18 Percent Ni Maraging Steel	70 C	15 May	5.6
		19 June	0
2014 Aluminum	Ambient	15 May	5.1
		20 June	4.0
2014 Aluminum	70 C	15 May	5.1
		19 June	0
2219 Aluminum	Ambient	15 May	4.0
		14 June	3.3
2219 Aluminum	70 C	15 May	0
			Not Analyzed

steel, 2219 aluminum, and 2014 aluminum tanks were being stored at ambient temperature. These tanks exhibited some decrease in FNO_2 concentration but remained above the minimum FNO_2 value for the INTO range. The 1018 carbon steel, 18 percent Ni (250 grade) maraging steel, 347 stainless steel, and 2014 aluminum tanks were being stored at 70 C. In all of these tanks, the FNO_2 content was essentially depleted. This indicated that these four materials did not passivate with INTO at 70 C under the conditions employed for the in situ formation of INTO from wet NTO.

The four tanks being stored at 70 C were replumbed into the fluorine addition system. The 2219 aluminum tank which had been on the fluorine manifold when the titanium tanks failed was also replumbed into the fluorine addition system. A total of approximately 13 pounds of fluorine was added to the five tanks. The contents of the tanks were analyzed for FNO_2 content and all were essentially within the INTO range, as shown in Table 2. All five tanks were placed in storage at 70 C. In June, the contents of the 347 stainless steel tank, which was being stored at ambient temperature, was transferred into another tank to be used on contract AF04(611)-11620 by direction of the Air Force Project Officer. The empty tank was then shipped to RPL, filled with GFP INTO, and returned to Rocketdyne where it was received on 3 August 1967.

Future Effort

All 10 tanks currently in storage will be periodically analyzed to determine their FNO_2 contents. The tanks are inspected at least three times per week to ensure that the proper environmental test conditions are maintained.

TABLE 2

RESULTS OF INFRARED ANALYSES AFTER FLUORINE ADDITION (2 AUGUST 1967)

Tank Material	Test Temperature, C	Mole Percent FNO_2
347 Stainless Steel	70	3.0
1018 Carbon Steel	70	3.5
18 Percent Ni (250 Grade) Maraging Steel	70	5.3
2014 Aluminum	70	4.0
2219 Aluminum	70	1.5

TASK II: TITANIUM STRESS CORROSION

SUMMARY

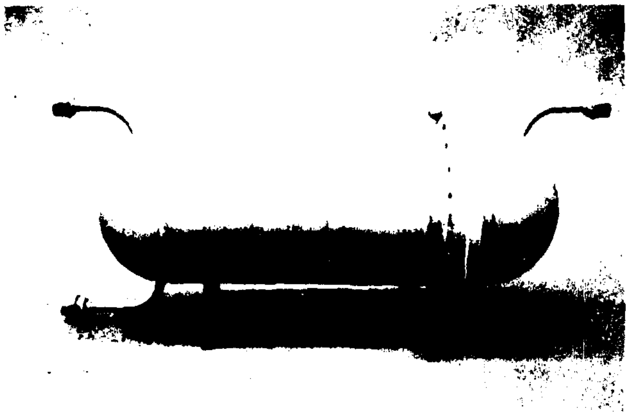
Titanium, 6Al-4V alloy stress corrosion specimens were exposed to NTO inhibited with FNO_2 at 70 C and at room temperature for a period of 43 days, both completely immersed, and half immersed in the liquid, at a tensile stress of 90,000 psi. No specimens failed during the test period. Tensile testing and metallographic examination revealed no evidence of deterioration due to the exposure, except for a uniform dissolution of 0.0005 to 0.001 inch of metal.

EXPERIMENTAL

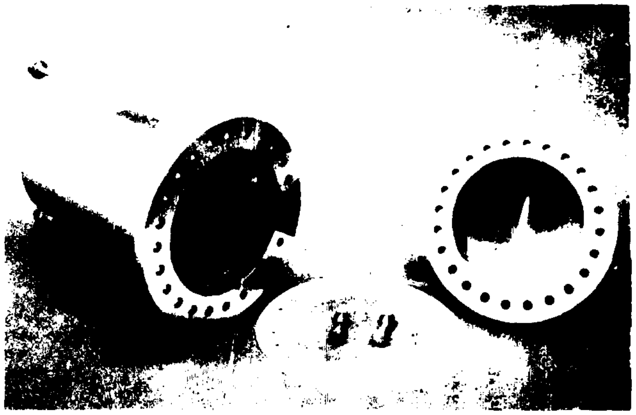
Tanks

Two tanks were constructed to hold the INTO. The design is shown in Fig. 2. The material was 6Al-4V titanium alloy throughout, except for the flare fittings which were stainless steel, the bolts (A286 alloy), and the Teflon gasket. The inside diameter was 6-1/4 inches, with hemispherical ends, and a 10-inch straight section between these. Fill, sampling, and vent tubes were installed as shown in Fig. 2B. Wall thickness was 0.63 inch.

The tanks were pressurized hydrostatically to 270 psig, maintained for 2 minutes at that pressure, and depressurized. This procedure was repeated five times. There was no leakage. The tanks were fabricated by TIG welding in a chamber containing an argon-helium atmosphere and were radiographically inspected to Rocketdyne Class I requirements (RA 0611-006). After welding, the parts were stress relieved for 3 hours at 1000 F, finish machined, and chemically cleaned.



A: Titanium tank for stress corrosion testing. Fill tube is at right, sampling at lower left, and vent at upper left. Tank was entirely titanium, except for the Teflon gasket, A286 bolts, and stainless-steel flare fittings.



B: Titanium stress corrosion test tank, disassembled. Bolts were tightened to 30 lb-ft torque. No leakage occurred.

Figure 2. Stress Corrosion Testing Tanks

Stress Corrosion Frames

The stress corrosion frames are shown in Fig. 3. They were made entirely of 6Al-4V titanium alloy. The frames were rough machined and then heat treated as follows:

1. Degreased in acetone
2. Solution treated in air at 1750 F for 1 hour, then water quenched for 4 seconds maximum.
3. Aged in air at 1000 F for 8 hours, then air cooled

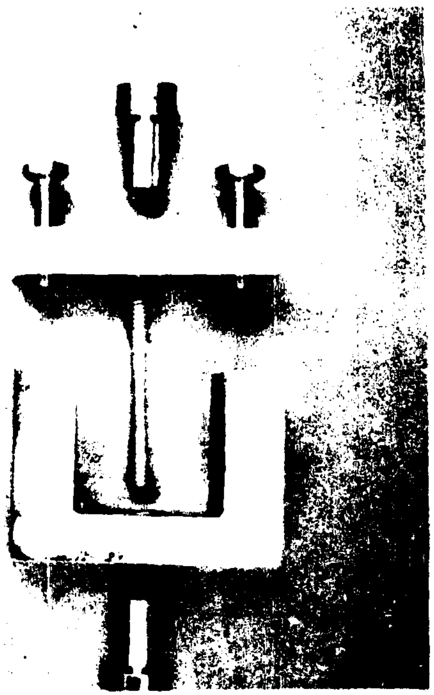
The frames were then finish machined, removing all surface contamination from the air, and then cleaned and packaged.

Specimen Stressing

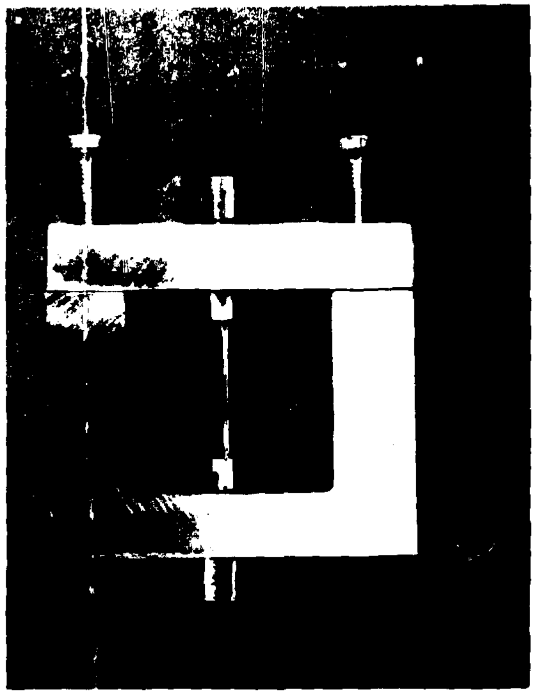
The specimens were to be loaded to 90,000 psi in tension during the test. Three specimens were selected at random. Each was stressed to 90,000 psi in a tensile machine and the extension under load recorded on a chart. This test was repeated three times for each specimen. From the nine tests, an average stress-strain slope of 12.2×10^6 psi and an average extension of 0.00523 in./in. were determined. Using the same extensometer, the specimens were loaded in the stress frames by torquing the cap screws to produce the required extension (Fig. 3).

Tank Loading

The loaded frames were cleaned in acetone and loaded into racks in groups of three, as shown in Fig. 4A. Two racks were loaded into each tank. These racks were positioned in the tanks at different heights supported on rails as shown in Fig. 4B. The volume of liquid to be



A



B

Figure 3. Titanium Stress-Corrosion Test Frames. (Specimen is threaded into the end nuts, which in turn fit into the frames. Stress is applied by tightening the cap-screws. Spherical-ends of the cap screws mate into spherical seats in the frame proper.)

A

B

Figure 4. Loading and Supporting of Stress Frames in Tanks

added was calculated to provide for complete immersion of one set of three and to position the liquid-gas interface in the middle of the specimens in the other set. After positioning the frames in the tank, the gasket was inserted and the bolts tightened uniformly to 30 lb-ft torque.

Three frames, with their stress specimens, were stored in a dessicator as controls. Other specimens were maintained unstressed at room temperature.

Specimen Preparation

The raw material was purchased from Titanium Metals Corporation of America as 1.0-inch bar stock conforming to AMS 4923B, mill annealed. Pertinent information concerning the material, as certified by the vendor, is presented in Tables 3 and 4.

The metallographic structure of the material as received, taken at the center of the 1.0 inch round, is shown in Fig. 5. The structure is well defined alpna-beta, fine grained, and very clean.

The 1.0-inch-diameter stock was abrasively cut into quarters, from which the specimens were to be made. After cutting, the material was heat treated as follows:

1. Cleaned in acetone
2. Vacuum annealed at 1400 F for 4 hours, then vacuum cooled to 800 F, and air cooled
3. Solution treated in air at 1750 F for 1 hour, then water quenched for 4 seconds maximum
4. Aged in air at 1000 F for 8 hours then air cooled

TABLE 3

VENDOR'S CERTIFICATION OF CHEMICAL ANALYSIS OF TEST BAR MATERIAL (HEAT #G1750)

ELEMENT	AMOUNT, percent
C	0.024
Fe	0.14
N	0.014
Al	6.4
V	4.2
H	0.005
O	0.12

TABLE 4

VENDOR'S CERTIFICATION OF MECHANICAL PROPERTIES OF TEST BAR MATERIAL (HEAT #G1750)

Yield Strength, psi	138,000
Tensile Strength, psi	143,500
Elongation, percent in 2 inches	20
Reduction of Area, percent	47
Notched time fracture 150,000 psi in 5 hours	
Elevated temperature properties, 1/2 hour at 700 F	
Yield Strength, psi	84,500
Tensile Strength, psi	101,300
Elongation, percent in 2 inches	19
Reduction of Area, percent	57
Beta Transus 1825 F to 1835 F	

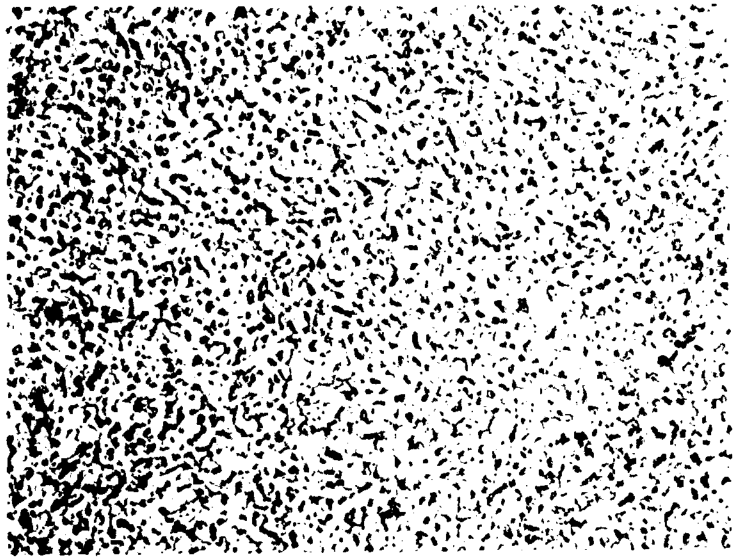


Figure 5. Structure of Specimen Material. (This photomicrograph is a cross-section through the as-received bar, 1.0 inch diameter, taken at the center; well defined alpha-beta, fine grained, and very clean; Keller's etchant; 500X.)

After heat treatment, a metallographic examination confirmed that no contamination had occurred that would not be eliminated during machining. The pieces were then finish-machined.

Inspection for Failures

The stress-corrosion tests were to run for 60 days. To determine whether any specimens had failed without opening the tanks, a radiographic technique was developed. An Iridium 192 pill was used, initially approximately 10 curies. By photographing horizontally through the tank with the film set vertically behind it, excellent resolution was obtained with a 3-minute exposure on Type A film at 3 feet. The lower portion of the frame is held in position by the specimen (Fig. 4B). In the event of failure, the frame would drop to the bottom of the tank, and this would be immediately evident on the radiograph.

RESULTS

The contents of the tanks were sampled to determine their FNO_2 contents at the start of the test and weekly thereafter. At all times, the FNO_2 concentrations remained above the lower limit of the INTO range. The results of the first and last analysis on both tanks are presented in Table 5.

TABLE 5

FNO_2 CONTENTS OF STRESS CORROSION TANKS

Tank	Test Temperature	FNO_2 Content, mole percent	
		26 May 1967	20 June 1967
1	70 C	5.8	1.9
2	Ambient	5.7	5.0

The stress corrosion tests were terminated on 6 July 1967 due to penetration of the two titanium 6Al-4V storage tanks reported in Task I. The 70 C tank was removed from the oven, but neither tank was cleared of INTO until 7 July 1967; there was a total of 43 days exposure. After venting out of the tanks, they were purged with dry nitrogen.

On 10 July 1967, the tanks were opened in a dry box. The condition of the interior is shown in Fig. 6. All titanium alloy surfaces were coated uniformly with a cream-colored layer, 0.010 inch thick. This coating was highly hygroscopic, and dissolved on absorbing moisture from the air releasing NO_2. In Fig. 6 this absorption process has already occurred. After flushing with water, the tank interior appeared smooth and no pitting was evident to the unaided eye. None of the stress corrosion specimens had broken. There was no apparent difference in the condition of any of the specimen groups, regardless of exposure situation. All were uniformly coated with the same cream-colored coating. Representative groups in several stages of coating dissolution are shown in Fig. 7. Again, the coating was approximately 0.010 inch thick.

The stress corrosion tanks were not as severely attacked by the preformed INTO as were the 24-gallon titanium tanks by INTO prepared by adding wet NTO and then bubbling in fluorine to form the INTO in situ. However, results on small bombs obtained during Task III indicate that titanium 6Al-4V will not passivate against even preformed INTO. Thus, the lesser attack noted in the stress corrosion tanks indicates only that the reaction was slower.

Measurements of specimen diameter after rinsing off the coating indicated that between 0.0005 and 0.001 inch of metal had been removed. The specimens were fitted with the same extensometer used in loading, and the load was released. All specimens contracted slightly more than they had originally been stretched, due to cross-sectional decrease by corrosion. The results of tensile tests on the 12 specimens are presented in Table 6. Results on six identical control specimens stored unloaded in air are presented in Table 7. It is evident that there was no significant deterioration of the

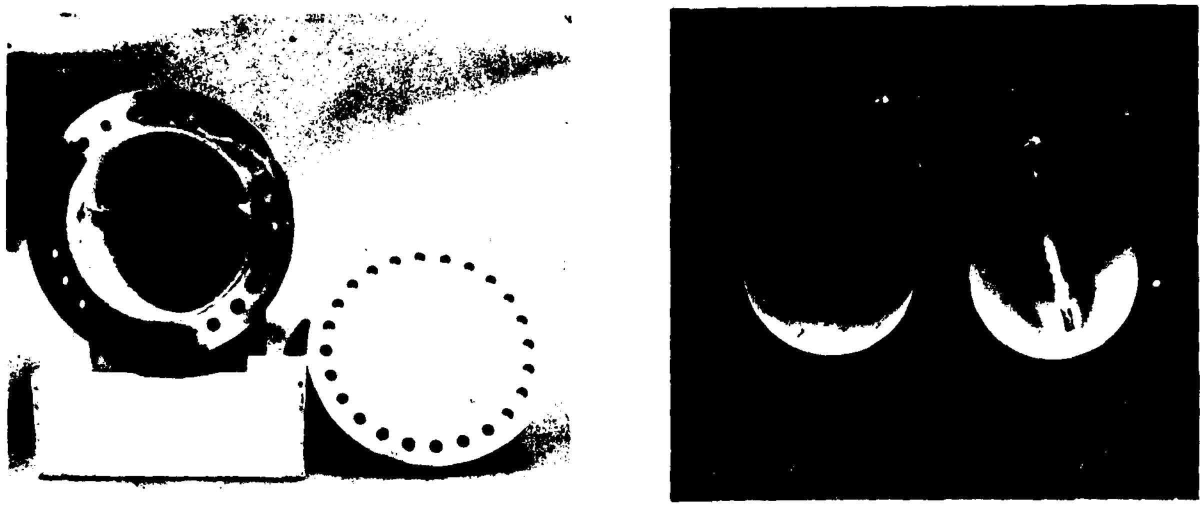
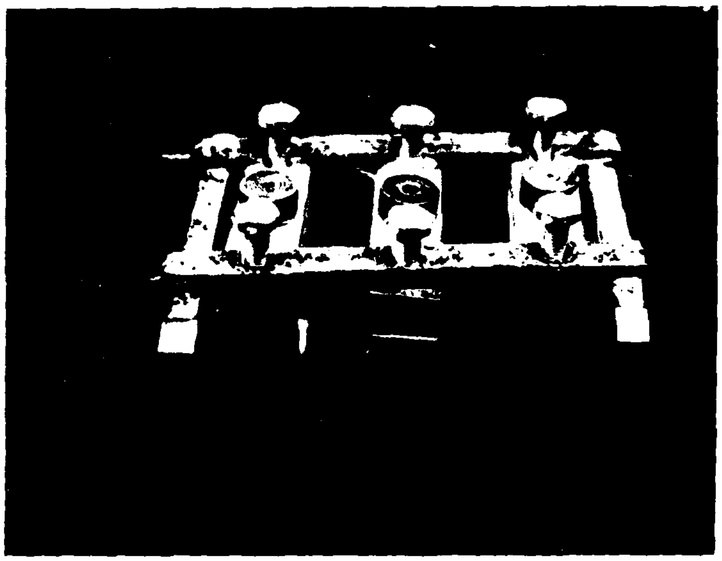


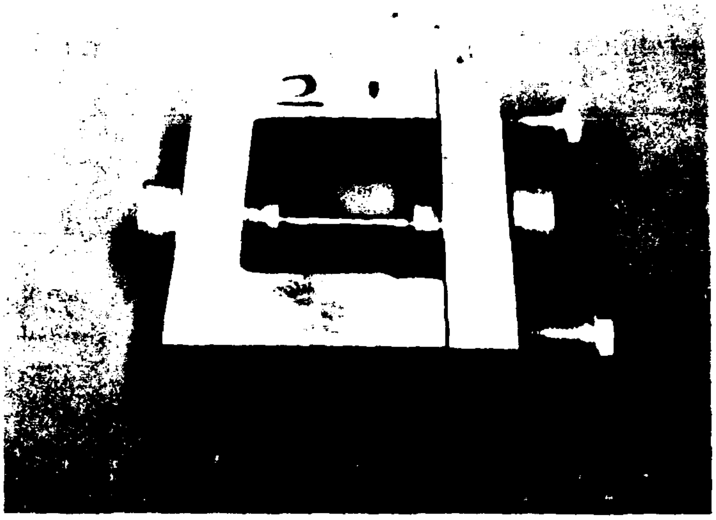
Figure 6. Condition of Tanks Immediately After Opening
(Water absorption from the atmosphere has
already begun.)

A. Immediately After Removal

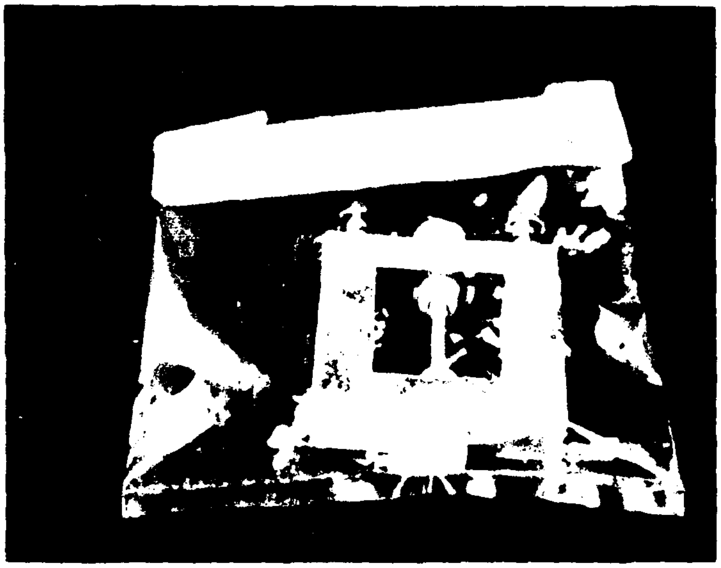


B. After 5 Minutes in Air

Figure 7. Frames and Specimens After Test



C. After Water Rinsing



D. Packaged Before Removal From Dry Box
to Exclude Air

Figure 7 (Concluded)

TABLE 6

MECHANICAL PROPERTY TESTS ON SPECIMENS AFTER EXPOSURE

Specimen No.*	Cross-Sectional Area, sq in.	Yield Strength (0.2 percent offset), psi	Ultimate Strength, psi	Elongation percent in 0.5 inch (0.5 inch = 4 diameters)
101	0.0121	157,800	171,000	16.0
102	0.0119	159,400	175,200	16.0
103	0.0117	159,200	176,000	14.0
Average		158,800	174,700	15.3
111	0.0117	166,800	179,200	14.0
112	0.0119	161,200	176,000	14.0
113	0.0119	161,000	174,000	14.0
Average		163,000	176,400	14.0
201	0.0125	156,000	172,500	14.0
202	0.0121	161,200	174,000	16.0
203	0.0121	160,700	177,000	16.0
Average		159,300	174,500	15.3
211	0.0119	160,000	179,400	14.0
212	0.0123	158,600	173,500	16.0
213	0.0121	161,000	174,000	14.0
Average		159,900	175,600	14.7

*First digit indicates exposure Temperature 1 = 70 C
2 = Room temperature

Second digit indicates exposure type 0 = Half immersed
1 = Fully immersed

Third digit indicates individual specimen

TABLE 7

MECHANICAL PROPERTIES OF CONTROL SPECIMENS

Specimen No.	Cross-Sectional Area, sq in.	Yield Strength (0.2 percent offset), psi	Ultimate Strength, psi	Elongation percent in 0.5 inch (0.5 inch = 4 diameters)
1	0.0121	159,800	176,200	14.0
2	0.0121	159,400	175,700	13.0
3	0.0125	160,300	176,300	14.0
4	0.0125	160,300	176,300	15.5
5	0.0121	157,300	175,200	16.0
6	0.0123	161,300	174,400	15.5
Average		159,700	175,700	14.7

specimens due to exposure to INTO during this series of tests. This is corroborated by Fig. 8, which shows a completely normal cup-and-cone ductile fracture, with no indication of embrittlement. Figure 9, at higher magnification, shows the typical alpha-beta structure of 6Al-4V alloy with no indication of localized chemical attack.

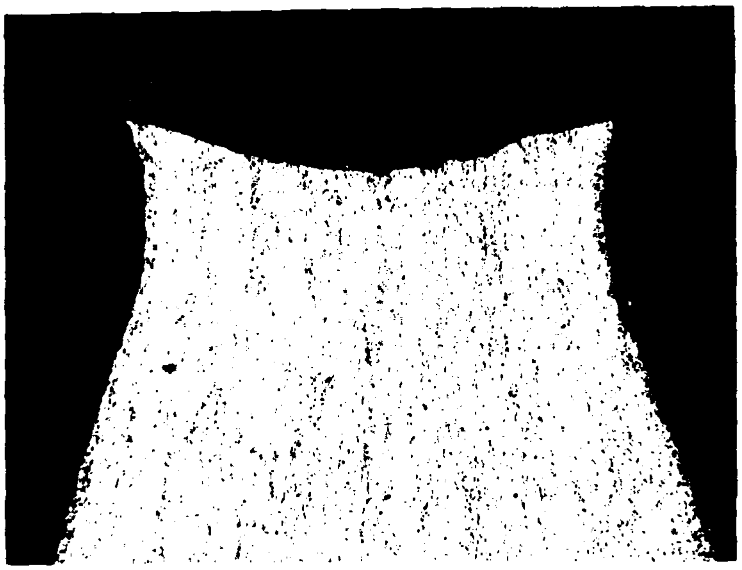


Figure 8. Specimen After Test at 50X Magnification (Structure is normal and ductile. No evidence of corrosion is present.)

Figure 9. Specimen After Test at 200X Magnification
(No evidence of localized chemical attack.)

TASK III: SMALL-BOMB STORABILITY

SUMMARY

Small-bomb storability tests are being conducted with INTO prepared from dry NTO (0.08 weight percent H_2O) and with INTO prepared from wet NTO (0.2 weight percent H_2O). The bombs (20 cc volume) are fabricated of two materials: (1) type 250 (18 percent Ni) maraging steel and (2) type A286 steel welded with Hastelloy-W rod. Identical tests on bombs constructed of titanium 6Al-4V were terminated because of the failure of the large titanium 6Al-4V tanks in which INTO was being stored during Task I of this program and the lack of storability of INTO in this alloy previously reported (Ref. 2 and 3) during this task. Storage tests at 130 C for 24 hours were completed and the results were reported previously (Ref. 3). Twelve-month tests are now in progress at ambient temperature and at 70 C. These tests are in their eighth month. The available data indicate that INTO is storable in A286 steel welded with Hastelloy-W rod, that it is not storable in titanium 6Al-4V, and that it is probably not storable in 250 maraging steel.

EXPERIMENTAL

The bombs were loaded according to the procedure described in the first quaterly progress report of this program (Ref. 4). All bombs were passivated for 96 hours at 70 C with a 30-mole percent solution of FNO_2 in NTO prior to the initiation of the tests. The FNO_2 concentrations used were above those of the INTO range (1 to 3 mole percent) because previous storability tests (Ref. 3) for 24 hours at 130 C had indicated that it might require a substantial amount of FNO_2 to passivate some of the bombs. Analyses for FNO_2 were conducted by infrared spectrophotometric techniques (Ref. 1). Data from the tests conducted at ambient temperature and at 70 C are summarized in Table 8.

TABLE 8

PARTIAL RESULTS OF 12-MONTH STORAGE TESTS

Bomb Material*	Original NTO Water Content, weight percent	Temperature, C	FNO_2 Content, mole percent		
			0 Months	4 months	7 months
250 Maraging Steel	0.2	70	12.5	0 (18.0)**	0 (12.0)**
	0.08	70	20.0	0 (21.9)**	0 (16.0)**
	0.2	Ambient	12.5	9.9	5.6
	0.08	Ambient	20.0	12.6	6.6
A286 Steel Welded With Hastelloy-W	0.2	70	12.5	8.1	8.0
	0.08	70	20.0	16.3	13.0
	0.2	Ambient	12.5	14.4	15.1
	0.08	Ambient	20.0	19.7	18.0
Titanium 6Al-4V	0.2	70	12.5	0 (18.0)**	0
	0.08	70	20.0	0 (21.9)**	0
	0.2	Ambient	12.5	2.2 (18.0)**	10.0
	0.08	Ambient	20.0	1.3 (21.9)**	7.1

*Bombs were passivated for 96 hours at 70 C with 30 mole percent FNO_2 in N_2O_4 prior to test.
**Bombs were reloaded to the FNO_2 concentrations noted parenthetically.

Examination of Table 8 reveals that A286 steel welded with Hastelloy-W rod affects the FNO_2 concentration least. At ambient temperature, the FNO_2 content in the A286 test bombs did not change after 7 months. At 70 C, it only dropped 20 to 30 percent during the first 4 months of testing and then remained essentially constant throughout the next 3 months. The 250 maraging steel test bombs exhibited the second best qualities. At ambient temperature, the FNO_2 content dropped 25 to 40 percent during the first 4 months of testing and an additional 40 to 50 percent during the following 3 months. At 70 C, the FNO_2 was completely depleted after 4 months and, after reloading, it was again completely depleted after 3 more months. The titanium 6Al-4V tests were least successful. After 4 months at ambient temperature, 90 to 95 percent of the FNO_2 was depleted and, after reloading, 45 to 70 percent of the FNO_2 was depleted after 3 additional months. The apparent slowing of depletion of FNO_2 is not significant because the absolute amounts of FNO_2 lost during the period between 4 and 7 months was only slightly less than that lost between 0 and 4 months. At 70 C, the FNO_2 content of the titanium 6Al-4V bombs was completely depleted during the first 4 months of testing and, after reloading, the FNO_2 content was again completely depleted after 3 months. The interiors of the titanium 6Al-4V test bombs exhibited considerable pitting when they were opened and washed out with water. The two 70 C 250 maraging steel test bombs were reloaded with INTO. The titanium 6Al-4V test bombs were emptied and removed from testing because of the failure of the large titanium 6Al-4V tanks discussed in the Large-Tank Storage section of this report and the discouraging results with this alloy obtained during this Task. The remaining test bombs will be sampled periodically during the final 4 months of these tests.

CONCLUSIONS AND FUTURE EFFORT

The results of the 70 C and ambient temperature tests indicate that INTO is storable in A286 steel welded with Hastelloy-W and that it is not storable in titanium 6Al-4V. It is expected that further testing will confirm these conclusions. Type 250 maraging steel appeared to occupy *a position between A286 steel and titanium 6Al-4V in reactivity towards* FNO_2. Previous results on storability of FNO_2 at 130 C in A286 and in 250 maraging steel indicated that INTO was storable in both of these alloys (Ref. 3). The results to date at 70 C and at ambient temperature indicate that 250 maraging steel will probably not passivate against reaction with FNO_2. Further testing should clarify the storability of INTO in 250 maraging steel.

SUMMARY

The Task I large tanks are now being stored under test conditions. The tanks are made of 347 stainless steel, 250 maraging steel, 1018 carbon steel, 2219 aluminum, and 2014 aluminum. The FNO_2 contents of the tanks being stored at 70 C had dropped to zero after 1 month of storage. Fluorine was added to these tanks to again bring their FNO_2 contents into the INTO range and they were returned to the 70 C oven. Both titanium 6Al-4V tanks failed near the end of the fluorine addition process. These failures, coupled with the small-bomb storability results on titanium 6Al-4V alloy obtained during Task III, definitely establish that INTO should not be stored in this material.

The Task II titanium stress corrosion tests have been completed. Titanium 6Al-4V alloy stress corrosion specimens were exposed to NTO inhibited with FNO_2 at 70 C and at room temperature for a period of 43 days, both completely immersed and half immersed in the liquid at a tensile stress of 90,000 psi. No specimens failed during the test period. Tensile testing and metallographic examination revealed no evidence of deterioration due to the exposure, except for a uniform dissolution of 0.0005 to 0.001 inch of metal.

The Task III 12-month ambient temperature and 70 C small-bomb storability tests are now in their eighth month. Chemical analyses performed during the past quarter continue to indicate that INTO is storable in A286 steel welded with Hastelloy-W rod and that it is not storable in titanium 6Al-4V. The data also indicate that INTO is probably not storable in 250 maraging steel. Tests of the storability of INTO in titanium 6Al-4V have been discontinued because the results indicating nonstorability are now considered conclusive.

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13. ABSTRACT This program is concerned with extending the engineering evaluation of the new storable liquid oxidizer INTO which is NTO inhibited with 1 to 3 weight percent FNO_2. Storability tests are being conducted on INTO at ambient temperature and at 70 C in aluminum and iron tanks of 10- to 18-gallon volume. Two 24-gallon titanium 6Al-4V tanks which were to be tested during this program failed while fluorine was being bubbled into them to form FNO_2. This indicates that INTO is not storable in titanium 6Al-4V when formed in situ. Titanium 6Al-4V specimens were uniaxially stressed to 90,000 psi and stored in INTO at ambient temperature and at 70 C for 43 days. Stress corrosion failure did not occur. This test has been discontinued because of the results with the 24-gallon titanium tanks and with the small titanium bombs. Small-bomb storability tests (20 milliliters) in A286 steel and in 250 maraging steel at ambient temperature and at 70 C are in their eighth month. Chemical analysis for FNO_2 content continues to indicate that INTO is storable in A286 steel and that it is probably not storable in 250 maraging steel. Titanium 6Al-4V was similarly tested for 7 months. The data indicate that INTO is definitely not storable in this alloy under the test conditions.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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Fluorine						
Stress Specimens						
Storability						
Titanium						
A286 Steel						
250 Maraging Steel						

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